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Indian Standard

IS 17182 : 2020

एक्स - रे विवर्तन के आधार पर अवशिष्ट तनाव का मापन

X-ray Diffraction Based Residual Stress Measurements

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Non-Destructive Testing Sectional Committee, MTD 21

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Non-Destructive Testing Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard applies for obtaining the residual stress measurement by employing X-ray diffraction method based on ‘Bragg’s law’.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

X-RAY DIFFRACTION BASED RESIDUAL STRESS MEASUREMENTS

1 SCOPE

This standard prescribes the procedure to be followed for measuring residual stresses present on surfaces of polycrystalline metallic components by using X-ray diffraction (XRD) technique. This procedure is intended for use along with the procedures mentioned in manuals provided by the manufacturers of X-ray diffraction systems.

2 PRINCIPLE OF TEST

X-ray diffraction based stress measurements related to the measurement of elastic lattice strains using X-rays. The stresses present in the material induce changes in the spacing of the lattice planes from their stress free value to a new value that corresponds to the magnitude of the applied or residual stress. Hence, the basic law governing the measurement of spacing of lattice planes is the Bragg's law which is given by:

$$N\lambda = 2d_{hkl}\,Sin\theta,$$

where,

λ = X-ray wavelength,

d_{hkl} = lattice spacing of crystal planes (h,k,l), and

2θ = diffraction angle.

The stress measurements here relate to the changes in the distance between certain lattice planes (hkl) which diffract meeting the diffraction criteria.

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply:

3.1 Residual Stresses — Residual stresses can be defined as those stresses that remain in a material or body after manufacture and processing in the absence of external forces or thermal gradients.

3.2 ψ — The angle between the diffraction vector and the normal to the surface of the component, where diffraction vector is the normal to the plane whose d-spacing is under investigation.

3.3 $\mathbf{E_{hkl}}$ **—** Elastic modulus of the material for the crystallographic direction normal to the lattice planes in which the strain is measured as specified by the Miller indices *(hkl)*.

4 MEASUREMENT PRINCIPLE

The brief description of fundamental relation between lattice spacing and stress in the specimen surface is given below. Figure 1 depicts the pictorial representation of the angles and principle stresses for plane stress elastic model. The XRD based stress measurement is confined to the surface of the sample. In the exposed surface layer, a condition of plane stress is assumed to exist. That is, distribution of stresses described by principal stresses σ_1 and σ_2 in the plane of the surface, and no stress is assumed perpendicular to the surface, σ_3=0. However, a strain component perpendicular to the surface ε_3 exists as a result of the Poisson's ratio (ν), contractions caused by the two principal stresses.

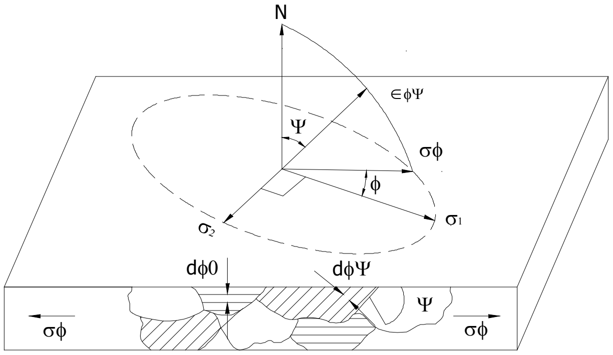

FIG. 1 PLANE STRESS ELASTIC MODEL

The expression of strain ($\varepsilon_{\Phi\Psi}$) in the sample surface at an angle Φ from the principle stress σ_1 is given by:

$$\varepsilon_{\Phi\Psi} = [\frac{1+\nu}{E}\sigma_{\Phi}\sin^2\Psi] - [(\frac{\nu}{E})(\sigma_1+\sigma_2)] \qquad - \quad (1)$$

Angle 'Φ' denotes the angle between one of the principal stress axes and the projection of the measured strain direction on the specimen surface. Angle 'ψ' is the angle between specimen normal and direction of strain measurement. E is Young's modulus, ν is Poisson's ratio.

Equation 1 relates the surface stress σ_{φ}, in any direction defined by the angle Φ, to the strain, $\epsilon_{\Phi\psi}$, in the direction (Φ,ψ) and the principal stresses in the surface. If $d_{\Phi\psi}$ is the spacing between the lattice planes measured in the direction defined by Φ and ψ, the strain ($\varepsilon_{\Phi\psi}$) can be expressed in terms of changes in the linear dimensions of the crystal lattice:

$$\varepsilon_{\Phi\Psi} = \frac{\Delta d}{d_0} = \frac{d_{\Phi\Psi} - d_0}{d_0} \qquad - \quad (2)$$

Where, d_0 is the stress free lattice spacing. From equations 1 and 2, we can write:

$$\frac{d_{\Phi\Psi} - d_0}{d_0} = [(\frac{1+\nu}{E})_{(hkl)} \sigma_\Phi \sin^2 \Psi] - [(\frac{\nu}{E})_{(hkl)} (\sigma_1 + \sigma_2)] \qquad - \quad (3)$$

where the elastic constants $(\frac{1+\nu}{E})_{(hkl)} and (\frac{\nu}{E})_{(hkl)}$ are the values for the crystallographic direction normal to the lattice planes in which the strain is measured as specified by the Miller indices (hkl).

The lattice spacing for any direction, then, is:

$$d_{\Phi\Psi} = [(\frac{1+\nu}{E})_{(hkl)} \sigma_\Phi d_0] \sin^2 \Psi - [(\frac{\nu}{E})_{(hkl)}$$

$$d_0 (\sigma_1 + \sigma_2)] + d_0 \qquad - \quad (4)$$

Equation 4 describes the fundamental relation between lattice spacing and the biaxial stresses in the surface of the sample. The lattice spacing is a linear function of the $\sin^2\Psi$. The intercept of the plot (d against.$\sin^2\Psi$) at $\sin^2\Psi = 0$ is:

$$d_{\Phi 0} = d_0 - (\frac{\nu}{E})_{(hkl)} d_0 (\sigma_1 + \sigma_2) =$$

$$d_0 [1 - (\frac{\nu}{E})_{(hkl)} (\sigma_1 + \sigma_2) \qquad - \quad (5)$$

which equals the unstressed lattice spacing, d_0, minus the Poisson's ratio contraction caused by the sum of the principal stresses. The slope of the plot (d against. $\sin^2\Psi$) is given by

$$\frac{\partial d_{\Phi\Psi}}{\partial \sin^2 \Psi} = (\frac{1+\nu}{E})_{(hkl)} \sigma_\Phi d_0 \qquad - \quad (6)$$

which can be solved for the stress σ_Φ:

$$\sigma_\varphi = (\frac{E}{1+\nu})_{(hkl)} \frac{1}{d_0} (\frac{\partial d_{\Phi\Psi}}{\partial \sin^2 \Psi}) \qquad - \quad (7)$$

where, $\frac{\partial d_{\Phi\Psi}}{\partial \sin^2 \Psi}$ is the slope of the plot d against $\sin^2 \Psi$.

Negative and positive slopes correspond to compressive and tensile stress respectively and the slope can be used to estimate σ_φ. This equation is a form of traditional X-ray diffraction based residual stress equation. It predicts a linear variation of d against $\sin^2\psi$. The stress in the σ_Φ direction may be obtained directly from the slope of a least square line fitted to experimental data measured at various ψ. If the elastic constants E, ν and the unstressed plane spacing d_0 are known. This procedure is known as $\sin^2\psi$ technique since it utilizes multiple ψ - tilts.

5 X-RAY DIFFRACTION BASED RESIDUAL STRESS MEASUREMENT SYSTEM

The XRD based residual stress measurement system available with the user may be installed and operated as per the operating manual provided by the supplier. The equipment should have facility to make multiple ψ angle measurements of the peak position. As the instruments used for stress measurements generally have a limited 2θ range in the high angle region (for example 2θ between 120 to 170°), suitable X-ray target material which would decide the X-ray wavelength and suitable crystal plane which would diffract in this angular range should be chosen.

6 TEST PROCEDURE

6.1 Reference Standards

6.1.1 *Peak 2θ Verification*

The goniometer's alignment can be verified using a stress free sample, such as a well annealed powder of lanthanum hexaboride or silicon or a quartz block. The diffractometer scans the output to define the position of the peaks which can be compared to known peaks on the JCPDS from the well-established database. The closeness of the results defines the instrument alignment.

6.1.2 *Material Constants*

The elastic constants $(1 + \nu/E)_{(hkl)}$ and $(\nu/E)_{(hkl)}$ of the material under investigation should be obtained from the well-established literature.

6.2 Preparation

6.2.1 *Sample Preparation*

Sample must be clean enough to get maximum reflection. Paints and other coating layers present on the location of test sample must be cleaned using proper solvents, such as acetone. If the above procedures do not give sufficient intensity from the material surface under examination, electro polishing has to be carried out using suitable chemicals.

6.2.2 *Target Selection*

Proper selection of wavelength should be done depending on the material to be examined by choosing the appropriate X-ray target so that the diffracted beam is measured within the 2θ range of the instrument for a specific (h, k, l) plane. Use K_β filter if K_α radiation is to be used for the particular material under investigation.

The elastic constants for the plane which is diffracting in the angular range of the instrument based on the wavelength of the radiation should be determined as per **6.1**.

6.2.3 *Sample Alignment*

The sample location is aligned such that the axis of the X-ray source and the detector matches with the top surface of the sample. Adjust the height of the goniometer with repect to the sample by operating the respective control switches. Make sure that the detector and the source are aligned along the required direction of the stress measurement.

6.3 Test Scan and Fixing the Parameters

Using the necessary software a test scan is run for any material. From the diffractogram obtained, the scan range, step width and dwell time are optimized and may be used for making the actual multiple ψ scans. After deciding the above parameters, X-ray tube details, current at the target, and ψ angles at which measurement should be carried out are fed into the automated system (software) provided by the manufacturer of the residual stress measurement system available with the user.

The operating voltage and current should be chosen appropriately to get well defined peaks from the planes which are being analysed. Normally, the operating voltage is fixed or at least above 20 kV. The X-ray intensity is normally recorded using a crystal detector making point by point measurements in the 2θ range or using a position sensitive detector which acquires data in the 2θ range simultaneously. It is conventional to acquire data over a 2θ range which is at least 5 times more than the full width at half maximum of the XRD peak of the selected (h, k, l) plane.

6.4 Method of Testing

After ensuring the alignment and software setup, switch on the X-ray system for carrying out residual stress measurements. The software acquires the intensity data over the 2θ range as specified and at different ψ angles of the goniometer. The peak locations are determined for each ψ angle using dedicated algorithms for peak determination present in the software.

Analysis of the data acquired is to be carried out for finding out the residual stress value. After proper smoothing, background corrections and considering errors in the measurement, a plot between $\sin^2\psi$ against d is made and the slope is calculated. The d_o obtained from the peak value at $\psi = 0$ and the slope is substituted equation – (7), for obtaining the residual stress at the measured location.

The experiment is repeated at the same location for repeatability of the results. The experiment is carried out on different locations of the sample as per the requirements of the user.

7 ACCEPTANCE STANDARD

The recording criteria and acceptance standard shall be subject to agreement between the contracting parties.

8 REPORTING OF TEST RESULTS

The test report of residual stress at a given location shall contain the following information:

a) X-ray target used, operating voltage and current;
b) Details of material under investigation;
c) The stress constant used for the material under investigation;
d) Scan location and direction of measurement;
e) 2θ peak determined in degrees;
f) Plot of $\sin^2\psi$ *versus* 2θ or d;
g) Residual stress in MPa with reliability; and
h) Personnel performing the examination and their qualifications and signatures.

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